CARBON NANOTUBE TECHNOLOGY FOR MICRO GAS CHROMATOGRAPH AND CHEMICAL NANOSENSORS

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Abstract

Multiwall carbon nanotubes (MWCNT), with number of wall layers in range of 5 - 30 and lengths up to 500 micrometers are synthesized by microwave plasma enhanced chemical vapor deposition (MPCVD) using evaporated multi-layer Fe catalyst. The growth of MWCNT inside a deep reactive ion etched (DRIE) Si channel of the pre-concentrator focuser (PCF) section of an on-chip micro gas chromatograph (μGC) is reported for the first time by applying the catalyst selectively in the channels. A MWCNT based chemical nanosensor has also been fabricated using the same technology and initial test results are reported.

CARBON NANOTUBE TECHNOLOGY FOR μGC CHANNELS

An intensive study of tube density, diameter and catalyst application led to the selection of Fe/Ti (10 nm/10nm) double layer, evaporated on p-type Si wafers, for growth of high quality multiwall carbon nanotubes (MWCNT). While Fe serves a catalyst, the Ti layer provides a diffusion barrier. The samples were treated in hydrogen plasma for 10 minutes at 40 torr using H_2 flow rate of 20 sccm before increasing the microwave power to 1800 W. After the hydrogen plasma treatment, the MWCNT were grown at 650 ^oC by introducing CH_4 at a flow rate of 10 sccm into the growth chamber. The growth rate was approximately 10 micro-meters per minute [3].

The preconcentrator channels were fabricated in Si by deep reactive ion etching (DRIE) as shown in Figure 1(a) followed by evaporation of Fe/Ti (10 nm/10nm) double layer at $5x10^{-6}$ torr. Photoresist, left at the top of the channels after DRIE, was used to lift off the unwanted metal layers on the top surface of Si channels. After a MWCNT growth time of 15 minutes in the channels the sample was cooled down to room temperature in vacuum [1] [2]. Figure 1(b-d) shows the sample after the growth. The tube length and density are approximately 50 μm and $4.2x10^9$ cm^{-2}, respectively. The TEM pictures (JEOL 2010F field emission TEM), indicating number of wall layers in the range of 5 – 10 and shown in Fig. 1(e-f), were taken on a separate set of samples.

CARBON NANOTUBE NANOSENSOR TECHNOLOGY

The sensor structure was fabricated on a quartz substrate using growth conditions described above. The Fe/Ti metal layer was patterned to form the electrodes separated by 200 μm. As the tubes are 400 μm tall, the tubes from the neighboring electrodes overlap to form the sensor structure as shown in Figure 2(a).

Sensor Testing

The fabricated sensor was placed on a temperature-controlled stage inside a high vacuum chamber kept at $2x10^{-7}$ torr. The surface temperature of the sensor structure was measured by a K type thermocouple. A Keithley 2010 multimeter was used to monitor the resistance change of the sensor and a HP 4140B pA meter/ DC voltage source was used to characterize the current-voltage (I-V) characteristics. Figure 2(b) shows the response of the sensor after exposure to acetone in air at room temperature, indicating shorter response times than those reported in the literature [4]. The resistance of the exposed sample decreases below that of the control sample. Figure 2(c) shows the I-V characteristics of the sensor structure in vacuum at 25 ^oC. The structure shows a linear current-voltage response which indicates there is no junction between the MWCNT and the metal electrode [5]. Figure 2(d) shows the resistance- temperature response of the sensor structure in vacuum. When the sample was heated up, the pressure inside the vacuum chamber increased from $2x10^{-7}$ torr to $1x10^{-6}$ torr. A resistance hysteresis was also observed during the temperature cycle. This phenomenon appeared even after 48 hours inside the vacuum chamber and two temperature cycles.

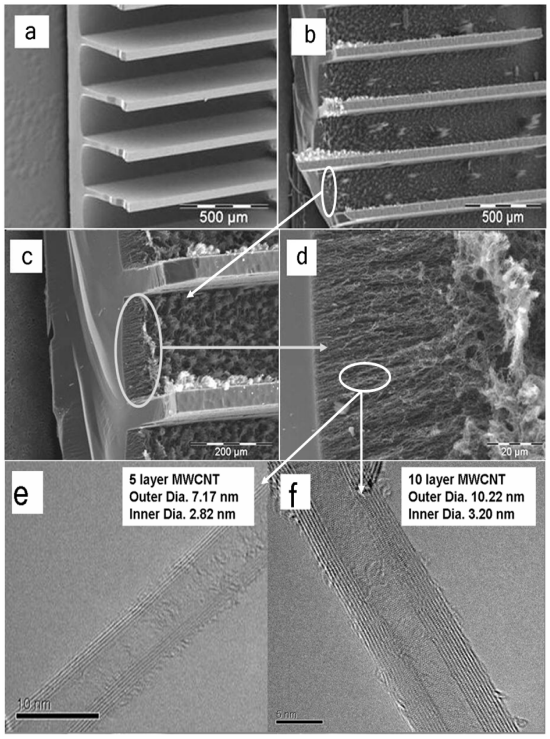

Figure* 1 *(a) - (d) SEM of the PCF; (e),(f) TEM of the MWCNT.

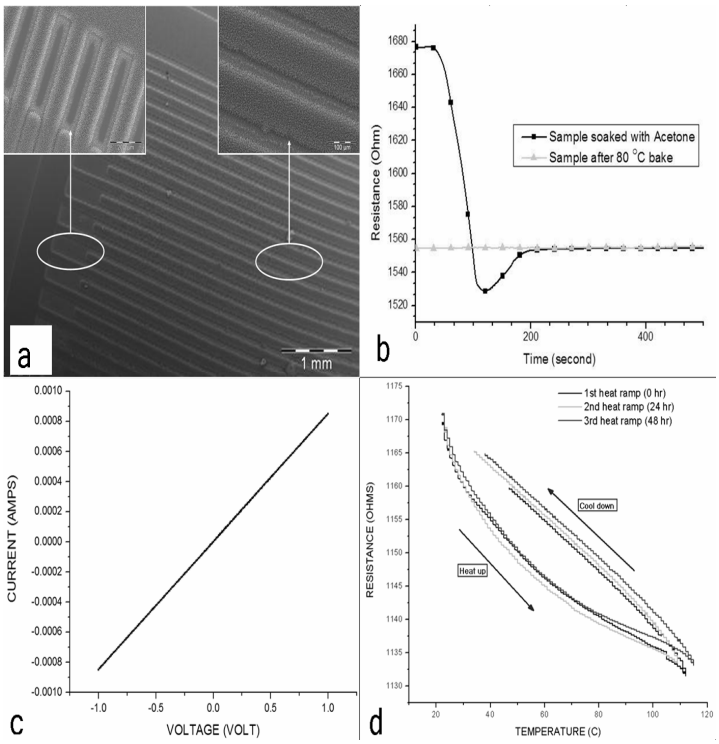

Figure* 2 *(a) SEM of the sensor; (b) Resistance measurement in air, (c) I-V characteristic, (d) Resistance-Temperature response of the sensor in vacuum.

REFERENCES

1. W. C. Tian, S. W. Pang, C. J. Lu, and E. T. Zellers "Microfabricated preconcentrator/focuser for a micro-scale gas chromatograph," J. MEMS 12(3), 264-272, (2003).
2. D.M. Aslam, K. Najafi, K.D. Wise and T. Zellers, "Poly-Carbon Nano- And Micro-Technologies For Wireless Integrated Micro-Systems", COMS 2002, Ypsilanti, MI 2002.
3. U. Kim, R. Pcionek, D.M. Aslam and D. Tománek, ”Synthesis of high-density carbon nanotube films by microwave plasma chemical vapor deposition”, Diamond & Related Materials (10)11 pp. 1947-1951 (2001).
4. L. Valentini, I. Armentano, J. M. Kenny，C. Cantalini，L. Lozzi and S. Santucci，” Sensors for sub-ppm NO2 gas detection based on carbon nanotube thin films”, Applied Physics Letters (82)6 pp. 961-963 (2003).
5. Jing Kong, Nathan R. Franklin, Chongwu Zhou, Michael G. Chapline, Shu Peng, Kyeongjae Cho, Hongjie Dai, “Nanotube Molecular Wires as Chemical Sensors”, Science (287) pp. 622-625 (2000).